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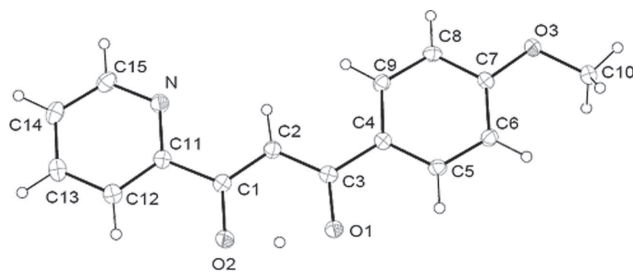
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Crystal structure of (Z)-3-hydroxy-3-(4-methoxyphenyl)-1-(pyridin-2-yl)prop-2-en-1-one, $C_{15}H_{13}NO_3$



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Abstract

$C_{15}H_{13}NO_3$, monoclinic, *Cc* (No. 9), $a = 3.9230(3)$ Å, $b = 26.578(2)$ Å, $c = 11.7147(12)$ Å, $\beta = 94.028(8)^\circ$, $V = 1218.4(2)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0387$, $wR_{ref}(F^2) = 0.0877$, $T = 173$ K.

CCDC no.: 1525410

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

To a suspension of sodium (0.4 g; 17.40 mmol) in anhydrous toluene, was added ethyl 2-picolinate (2 g; 13.28 mmol) in toluene. Then 1-(4-methoxyphenyl) ethanone (1.99 g;

Table 1: Data collection and handling.

Crystal:	Colourless platelet
Size:	$0.50 \times 0.15 \times 0.06$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.0 cm^{-1}
Diffractometer, scan mode:	Xcalibur Sapphire3, ω -scans
$2\theta_{\max}$, completeness:	58.6° , $>87\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6523, 2714, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2410
$N(\text{param})_{\text{refined}}$:	181
Programs:	CrysAlis ^{PRO} [8], SHELX [9], ORTEP [10]

13.28 mmol) was added at 0 °C. The resulting mixture was stirred at room temperature for 7 days. The precipitate formed was filtered, washed and dissolved in water. The neutralized solution was extracted with CH_2Cl_2 , and the organic layer was dried and concentrated *in vacuo*. The obtained residue was chromatographed on silica using $\text{CH}_2\text{Cl}_2/\text{MeOH}$ as eluant to give the desired product. Crystals of the title compound were obtained from hot ethanol by slow evaporation. Yield: 33%, *M.p.* 112–114 °C.

Experimental details

The structure has been first solved in a triclinic unit cell of parameters $a = 7.85$, $b = 11.72$, $c = 13.43$ Å, $\alpha = 89.4$, $\beta = 81.6$, $\gamma = 86.0^\circ$. The unit cell contains four independent molecules. Final refinement in these conditions with anisotropic displacement parameters led to an agreement factor $R1 = 4.59\%$. Except the freely refined H atoms that display rather high standard deviations for which positions are not precisely determined, the four molecules are symmetry-related and it became obvious that the cell must be transformed to the monoclinic cell described here ($-0.5 \ 0 \ 0 \ -0.5 \ 0 \ 2 \ 0 \ 1 \ 0$). Final refinement in the monoclinic space group improves the agreement factor to 3.87%. The acidic hydrogen atom is placed between O1 and O2 atoms. This would indicate that this H atom is easily exchangeable between the two oxygen atoms.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	-0.1882(5)	-0.36669(8)	-0.2671(2)	0.0259(5)
C2	-0.2952(6)	-0.36593(9)	-0.3825(2)	0.0267(6)
C3	-0.2108(5)	-0.40601(8)	-0.4539(2)	0.0249(5)
C4	-0.3192(6)	-0.40770(8)	-0.5771(2)	0.0241(5)
C5	-0.2310(6)	-0.44855(8)	-0.6429(2)	0.0266(5)
H5	-0.1015	-0.4752	-0.6074	0.032*
C6	-0.3270(5)	-0.45148(9)	-0.7591(2)	0.0269(6)
H6	-0.2665	-0.4799	-0.8024	0.032*
C7	-0.5126(5)	-0.41221(9)	-0.8110(2)	0.0246(5)
C8	-0.6015(6)	-0.37083(9)	-0.7473(2)	0.0282(6)
H8	-0.7275	-0.3440	-0.7834	0.034*
C9	-0.5073(5)	-0.36858(9)	-0.6318(2)	0.0268(5)
H9	-0.5705	-0.3402	-0.5888	0.032*
C10	-0.5131(6)	-0.45106(9)	-0.9946(2)	0.0320(6)
H10A	-0.6025	-0.4831	-0.9682	0.048*
H10B	-0.2629	-0.4523	-0.9901	0.048*
H10C	-0.5990	-0.4451	-1.0741	0.048*
C11	-0.2740(6)	-0.32595(8)	-0.1876(2)	0.0267(5)
C12	-0.1731(6)	-0.32924(9)	-0.0722(2)	0.0316(6)
H12	-0.0481	-0.3576	-0.0427	0.038*
C13	-0.2582(7)	-0.29035(10)	-0.0005(2)	0.0368(6)
H13	-0.1917	-0.2915	0.0790	0.044*
C14	-0.4396(6)	-0.25025(10)	-0.0461(2)	0.0357(6)
H14	-0.5014	-0.2231	0.0010	0.043*
C15	-0.5309(7)	-0.25009(10)	-0.1626(3)	0.0360(6)
H15	-0.6576	-0.2222	-0.1934	0.043*
H1	-0.435(7)	-0.3384(10)	-0.409(2)	0.032(7)*
H2	0.019(11)	-0.4269(17)	-0.312(4)	0.099(14)*
N	-0.4521(5)	-0.28662(7)	-0.23402(19)	0.0314(5)
O1	-0.0315(4)	-0.44320(6)	-0.41288(16)	0.0320(4)
O2	-0.0054(5)	-0.40355(6)	-0.22220(17)	0.0348(4)
O3	-0.6225(4)	-0.41111(6)	-0.92382(15)	0.0310(4)

Discussion

Compounds incorporating a β -ketoenol moiety have yielded clinical integrase inhibitor drug candidates [1]. Tomassini et al. reported a series of β -ketoenol derivatives as effective inhibitors drug of influenza viral replication in both *in vitro* cell culture replication assays and *in vivo* mouse challenge model, without exhibiting any cytotoxicity [2–4]. Furthermore, complexes with β -ketoenols have received more attention in the area of medicinal chemistry [5], exhibiting anticancer activity against several tumoral cells strains [6], and cytotoxic activity in a chronic myelogenous leukemia cell line [7]. Herein, we report the preparation of a new pyridine incorporating a β -ketoenol moiety: (*Z*)-3-hydroxy-3-(4-methoxyphenyl)-1-(pyridin-2-yl) prop-2-en-1-one in moderate yield.

The crystal structure shows that the oxygen atoms share the same hydrogen atom placed just in between O1 and O2. This is due to the fact that each of the two atoms O1 and O2 alternately ensure the enole and ketone functions, and

are involved in a tautomer effect as reflected by the close distances C3–O1 1.287(3) and C1–O2 1.303(3) Å. The C4, C5, C6, C7, C8 and C9 atoms are in the same plane. The torsion angle O1 C3 C4 C9 of 178.2(2)° indicates that the benzene ring undergoes slight inclination with respect to the C3–C4 bond. Also the 2-pyridinyl moiety are in the same plane, while the torsion angle O2–C1–C11–N of –177.5(2)° indicate that the plane of pyridine ring undergoes also a slight inclination relative to plane of the rest of the molecule.

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